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## FINE GRAIN DENSE FERRITES

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REPORT NO. 7

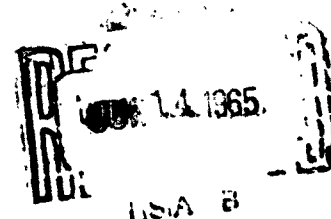
Continuation of contract DA36-039-AMC-00098(E)

Contract No. DA28-043-AMC-00178 (E)

DA Project No. ICO 24401 A 112

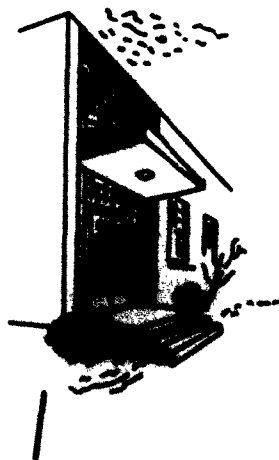
Third Quarterly Progress Report

1 January 1965 - 31 March 1965



U.S. Army Electronic Laboratories

Fort Monmouth, New Jersey



RAYTHEON COMPANY

Research Division

Waltham, Massachusetts 02154

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**FINE GRAIN DENSE FERRITES**

**Report No. 7**

**Continuation of Contract DA36-039-AMC-00098(E)**

**Contract No. DA28-043-AMC-00178(E)**

**Tech. Req. No. SCL-2101 P 18 February 1963**

**DA Project No. ICO 24401 A112**

**Third Quarterly Report**

**1 January 1965 - 31 March 1965**

**Object**

**To develop fine-grain dense ferrites  
for use at microwave frequencies**

**Prepared by**

**A. E. Paladino, J. S. Waugh, J. J. Green,  
J. Q. Owen, A. E. Booth, and M. J. Armstrong**

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## 1.0 PURPOSE

The purpose of the work performed under this contract is to develop and refine the techniques of preparation and evaluation of fine-grained, dense ferrites for use at microwave frequencies. Specifically, the applications are related to the improved high-power characteristics that result from decreasing the grain size of polycrystalline ferrites.

## 2.0 ABSTRACT

A number of new compositions were prepared in the vicinity of the compensation point in the series  $\text{Ni}_{1-x}\text{Co}_x\text{Fe}_2\text{O}_4$ , with  $0 \leq x \leq 0.04$ . No systematic variation in the threshold field or magnetic loss was observed with cobalt concentration, although threshold fields were as high as 108 Oe at KU-band. Lowering the hot-pressing temperature from 1200° to 1150°C had no consistent effect on microwave properties. Annealing studies at 1200° and 1250°C indicate that the threshold field decreases significantly with time for nickel and nickel-manganese ferrites, whereas it remains high in nickel-cobalt ferrites. The magnetic loss in general decreases with increasing time; it decreases most for nickel and nickel-manganese ferrites, and least for nickel-cobalt ferrites.

A satisfactory device geometry has been established for a differential phase-shift circulator. Low power phase shift and insertion loss measurements have been made on a conventional and fine-grain nickel-cobalt ferrite. Although the insertion loss of the fine-grain ferrite is about double that of the conventional material due to a magnetic loss of approximately twice that of the conventional material, a circulator with  $\leq 0.5$  dB loss can be constructed.

### 3.0 PUBLICATIONS, CONFERENCES, LECTURES, REPORTS

#### 3.1 Publications

None

#### 3.2 Conferences

On February 2, 1964, a meeting was held at USAERDL, Fort Monmouth, New Jersey, to discuss progress on the present program and the device testing to be made on fine-grain ferrites. Present from Raytheon were A. Paladino, J. S. Waugh, J. Green, E. Schlömann, J. Owen, and Al. Booth; and from the Signal Corps, R. Babbitt, I. Bady, and W. Malinofsky.

#### 3.3 Lectures

None

#### 3.4 Reports

None

## 4.0 FACTUAL DATA

### 4.1 Introduction

During the second quarter, considerable progress was made in preparing fine-grain ferrites with high  $h_{crit}$  for subsidiary absorption, low dielectric losses, and narrow ferromagnetic resonance linewidths. Values of the latter two properties on hot-pressed ferrites compared favorably with conventionally prepared ferrites, whereas the  $h_{crit}$  for subsidiary absorption were as much as five times greater than conventional materials. However, conventionally prepared ferrites had, in general, magnetic losses as much as four times lower than the hot-pressed materials. There were indications in the second quarter that annealing would lower the magnetic losses of the hot-pressed materials, and in some cases, not lower  $h_{crit}$ . It therefore appeared that some combination of hot pressing and annealing could be found that would result in a low loss, high-power ferrite material. Attention was given to this possibility during the third quarter.

Because of the optimistic outlook for fine-grain ferrites, the decision was made to start designing and building a phase shift section for testing the fine-grain materials, and establish a correlation between cavity and device measurements.

### 4.2 Preparation of Materials

The materials preparation procedure was the same as that outlined in the Second Quarterly Progress Report.

### 4.3 Hot Pressing

The hot-pressing procedure was the same as that reported in the Second Quarterly Progress Report. At 1200°C, the materials were pressed for 15 minutes, and at 1150°C they were pressed for 20 minutes.

#### 4.4 Property Measurements

Procedures for property measurements have been outlined in the Second Quarterly Progress Report.

#### 4.5 Results and Discussion

##### 4.5.1 New compositions

A number of new compositions were prepared this quarter, and are listed in Table I together with their properties. The first material is a nickel ferrite that was started last quarter and completed this quarter. The next seven are a series of cobalt compositions around the compensation point, and were made in an attempt to find some combination of grain size, cobalt content, and heat treatment to yield a high  $h_{crit}$  and low magnetic loss. Annealing studies on this series will be started this quarter.

There does not appear to be any systematic variation in  $h_{crit}$  or  $\chi''$  with cobalt content. In general, the properties are remarkably reproduced; the densities are all in excess of 98 percent with the exception of one sample (9696-38-4),  $h_{crit}$ 's are all in excess of 62 Oe, and the loss tangents are below  $5 \times 10^{-4}$ . The magnetic losses,  $\chi''$ , range between 0.027 and 0.040, which compare to values of between 0.007 and 0.014 for a good conventional material. These values for the fine-grain materials are significantly lower than had been obtained earlier in this program. (The requirements for  $\chi''$  in a device configuration will be discussed below.) Annealing should improve the magnetic losses as discussed below.

The next two compositions were prepared in large quantity (300 g) for future hot pressing and annealing studies that might be planned. Several times in the past extended series of experiments on a given powder have not been possible due to the small quantity originally made (about 100 g). Also, a large quantity of material may eventually be needed for the final device

TABLE I  
PROPERTIES OF FINE-GRAIN NICKEL FERRITE, NICKEL-COBALT FERRITE, AND  
NICKEL-MANGANESE FERRITE

| Code        | Composition  | Density and<br>Percent<br>of Theoretical | Grain<br>Size<br>( $\mu$ ) | $\kappa'$<br>X-Band | $\tan \delta$<br>X-Band | $h_{crit}$<br>(Oe)<br>KU-Band | $\chi''_{min}$<br>KU-Band | Remarks        |
|-------------|--|--|----------------------------|---------------------|-------------------------|-------------------------------|---------------------------|----------------|
| 9696-5-7E   | NiFe <sub>2</sub> O <sub>4</sub>                                       | 5.36<br>99.6                             | 5.0                        | 14.1                | $1.3 \times 10^{-4}$    | 24                            | 0.018                     |                |
| 9696-38-1   | Ni <sub>0.99</sub> Co <sub>0.01</sub> Fe <sub>2</sub> O <sub>4</sub>   | 5.34<br>99.5                             | 3.0                        | 13.7                | $4.0 \times 10^{-4}$    | 78                            | 0.028                     |                |
| 9696-38-2   | Ni <sub>0.985</sub> Co <sub>0.015</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.27<br>98.3                             | 3.4                        | 13.5                | $1.0 \times 10^{-4}$    | 62                            | 0.027                     |                |
| 9696-38-3   | Ni <sub>0.98</sub> Co <sub>0.02</sub> Fe <sub>2</sub> O <sub>4</sub>   | 5.35<br>99.9                             | 3.0                        | 13.9                | $5.5 \times 10^{-4}$    | 74                            | 0.030                     |                |
| 9696-5-8-1A | Ni <sub>0.973</sub> Co <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.31<br>99.1                             | 3.4                        | 13.7                | $5.0 \times 10^{-4}$    | 108                           | 0.030                     |                |
| 9696-38-4   | Ni <sub>0.97</sub> Co <sub>0.03</sub> Fe <sub>2</sub> O <sub>4</sub>   | 5.13<br>95.6                             | 2.0                        | 12.2                | $2.3 \times 10^{-4}$    | 98                            | 0.033                     |                |
| 9696-38-5   | Ni <sub>0.965</sub> Co <sub>0.035</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.33<br>99.4                             | 4.8                        | 14.2                | $2.0 \times 10^{-4}$    | 88                            | 0.034                     |                |
| 9696-38-6   | Ni <sub>0.96</sub> Co <sub>0.04</sub> Fe <sub>2</sub> O <sub>4</sub>   | 5.36<br>99.9                             | 3.2                        | 13.9                | $2.3 \times 10^{-4}$    | 98                            | 0.040                     |                |
| 9696-47-1A  | Ni <sub>0.973</sub> Co <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.35<br>99.8                             | 7.4                        | 14.1                | $3.0 \times 10^{-4}$    | 77                            | 0.033                     | 300 g<br>batch |
| 9696-47-2A  | Ni <sub>0.973</sub> Mn <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.34<br>99.4                             | 10.0                       | 14.2                | $6.0 \times 10^{-4}$    | 12                            | 0.011                     | 300 g<br>batch |

construction. The manganese containing sample will be hot pressed again to reduce the grain size and increase  $h_{crit}$ . It appears that above about  $10\mu$  for this composition,  $h_{crit}$  has reached a minimum.

#### 4.5.2 Effect of hot pressing temperatures

Several compositions hot pressed at  $1200^{\circ}\text{C}$  for 15 minutes last quarter were re-pressed at  $1150^{\circ}\text{C}$  for 20 minutes this quarter, and their properties are included in Table II. It was expected that at lower temperatures small grain sizes would have been obtained, with a resulting increase in  $h_{crit}$ . This expectation was not realized, and there does not appear to be any consistent dependence of  $h_{crit}$  on hot pressing temperature, at least for this series. In several cases, the dielectric loss tangent was improved with the lower hot pressing, whereas  $\chi''$  was independent of hot pressing temperature. The densities in all cases were in excess of 96 percent at  $1150^{\circ}\text{C}$ , and 98 percent at  $1200^{\circ}\text{C}$ . Pressing at the lower temperature does have the advantage of extending die life.

Certain generalizations can be made from the data in Tables I and II. First of all, there does not appear to be any obvious relationship between grain size and  $h_{crit}$  below about  $5\mu$ ; admittedly, the grain size variation is not great but at any constant value of grain size,  $h_{crit}$  can vary by a factor of two. It may be that a simple measurement of grain size is not a sufficient description of the microstructure, and perhaps some measure of the grain size distribution might be appropriate. Unfortunately a grain size distribution is not easily obtained, although some attempts will be made to determine the grain size distribution. Secondly, magnetic losses are higher than conventional materials by an amount that may ultimately limit the usefulness of fine-grain materials, especially where  $\chi''$  is greater than about 0.030. Finally, it appears possible to obtain threshold fields as high as 100 Oe, but more typically in the 50 to 80 Oe range.

TABLE II  
COMPARISON OF PROPERTIES OF FINE-GRAIN NICKEL, NICKEL-COBALT, AND  
NICKEL-MANGANESE FERRITES HOT PRESSED AT 1150°C AND 1200°C

| Code      | Composition                   | Temp<br>(°C) | Density and<br>Percent<br>of Theoretical | Grain<br>Size<br>( $\mu$ ) | $\kappa'$<br>X-Band | $\tan \delta$<br>X-Band | $h_{crit}$<br>(Oe)<br>KU-Band | $\chi''_{min}$<br>KU-Band | Remarks                  |
|-----------|-------------------------------|--------------|--|----------------------------|---------------------|-------------------------|-------------------------------|---------------------------|--------------------------|
| 9696-4-1  | $NiFe_2O_4$                   | 1150         | 5.37<br>99.8                             | 2.5                        | 14.7                | $1.3 \times 10^{-3}$    | 28                            | 0.017                     | Hot Press Time<br>20 min |
|           |                               | 1200         | 5.36<br>99.6                             | 2.9                        | 14.4                | $6.5 \times 10^{-3}$    | ---                           | ---                       | Hot Press Time<br>15 min |
| 9696-5-1  | $NiFe_2O_4$                   | 1150         | 5.19<br>96.5                             | 2.0                        | 13.2                | $5.0 \times 10^{-2}$    | 63                            | 0.025                     | ---                      |
|           |                               | 1200         | 5.31<br>98.7                             | 3.0                        | 11.3                | $1.9 \times 10^{-2}$    | ---                           | ---                       | ---                      |
| 9696-5-7  | $NiFe_2O_4$                   | 1150         | 5.32<br>98.9                             | 3.0                        | 11.8                | $2.4 \times 10^{-4}$    | 49                            | 0.019                     | ---                      |
|           |                               | 1200         | 5.35<br>99.4                             | 2.7                        | 14.4                | $1.8 \times 10^{-4}$    | 31                            | 0.019                     | ---                      |
| 9696-6-10 | $NiFe_2O_4$                   | 1150         | 5.26<br>97.8                             | 2.5                        | 13.9                | $4.0 \times 10^{-4}$    | 62                            | 0.020                     | ---                      |
|           |                               | 1200         | 5.26<br>97.8                             | 3.7                        | 13.4                | $9.7 \times 10^{-4}$    | 55                            | 0.020                     | ---                      |
| 9696-1-3  | $Ni_{0.973}Mn_{0.027}Fe_2O_4$ | 1150         | 5.21<br>96.8                             | 3.0                        | 13.7                | $2.7 \times 10^{-4}$    | 56                            | 0.025                     | ---                      |
|           |                               | 1200         | 5.36<br>99.6                             | 3.6                        | 14.6                | $3.4 \times 10^{-4}$    | 62                            | 0.018                     | ---                      |

TABLE II (Cont'd)

| Code      | Composition                   | Temp<br>(°C) | Density and<br>Percent<br>of Theoretical | Grain<br>Size<br>( $\mu$ ) | $\kappa'$<br>X-Band | $\tan \delta$<br>X-Band | $h_{crit}$<br>(Oe) |         | $\chi''$<br>min<br>KU-Band | Remarks |
|-----------|-------------------------------|--------------|--|----------------------------|---------------------|-------------------------|--------------------|---------|----------------------------|---------|
|           |                               |              |  |                            |                     |                         | KU-Band            | KU-Band |                            |         |
| 9696-5-6  | $Ni_{0.973}Mn_{0.027}Fe_2O_4$ | 1150         | 5.34<br>99.2                             | 3.0                        | 13.5                | $3.0 \times 10^{-4}$    | 62                 | 0.026   | ---                        | ---     |
|           |                               | 1200         | 5.38<br>99.9                             | 3.3                        | 13.9                | $1.3 \times 10^{-4}$    | 73                 | 0.029   | ---                        | ---     |
| 9696-6-12 | $Ni_{0.973}Mn_{0.027}Fe_2O_4$ | 1150         | 5.31<br>98.7                             | 2.0                        | 13.5                | $1.7 \times 10^{-4}$    | 70                 | 0.026   | ---                        | ---     |
|           |                               | 1200         | 5.29<br>98.4                             | 2.4                        | 13.7                | $1.5 \times 10^{-4}$    | 49                 | 0.015   | ---                        | ---     |
| 9696-5-5  | $Ni_{0.973}Co_{0.027}Fe_2O_4$ | 1150         | 5.20<br>96.7                             | 2.0                        | 14.0                | $1.6 \times 10^{-4}$    | 112                | 0.030   | ---                        | ---     |
|           |                               | 1200         | 5.38<br>99.9                             | 4.1                        | 14.6                | $3.4 \times 10^{-4}$    | 78                 | 0.027   | ---                        | ---     |
| 9696-5-8  | $Ni_{0.973}Co_{0.027}Fe_2O_4$ | 1150         | 5.35<br>99.4                             | 3.5                        | 14.6                | $1.7 \times 10^{-2}$    | 44                 | 0.026   | ---                        | ---     |
|           |                               | 1200         | 5.37<br>99.7                             | 3.0                        | 14.0                | $3.0 \times 10^{-3}$    | 87                 | ---     | ---                        | ---     |

#### • 4. 5. 3 Annealing studies

During the last quarter, several materials were hot pressed and also fired conventionally at temperatures low enough to produce grain sizes below  $6.0\mu$ . It was found that the magnetic losses were lower for the conventionally fired materials, which is probably related to the longer firing times used in this procedure. Magnetic losses are thought to be caused by local fluctuations in the saturation magnetization that are the result of chemical inhomogeneities. These inhomogeneities can be reduced by diffusion, and therefore  $\chi''$  should decrease when homogeneity is improved via heat treatments of extended duration. There are of course other subtleties involved in magnetic loss, but this is perhaps the most important consideration that bears on the current problem.

A number of materials were selected for the annealing studies, and are listed in Table III. These were selected on the basis of reasonably high  $h_{crit}'s$ , which in all cases is above 55 Oe. The last sample (9696-10FS) in the table was flame sprayed and hot pressed, and the one above it (9696-5-5) was the first sample selected for device testing.

The results of the annealing studies obtained thus far are included in Table IV. The blanks in this table will be filled during the final quarter of the program. The threshold fields were determined first to establish the limits of temperature and time within which  $h_{crit}$  remained relatively high, after which magnetic loss measurements were made. These limits appear to be somewhat different for each composition studied, being widest for the cobalt series, and less for the other two. The critical fields for nickel ferrite and nickel-manganese ferrite appear to be extremely sensitive to annealing being reduced to about half their original values after a 300-minute anneal. In contrast, the critical fields for the cobalt samples remain high and very close to their original values. Magnetic loss data are not complete enough to see any significant trends. One result is very encouraging, however. The  $\chi''$  for one of the cobalt samples (8354-76-7) has been reduced from 0.025 to 0.011 at  $1200^{\circ}C$  after a 60-minute anneal

TABLE III  
PROPERTIES OF FINE-GRAIN NICKEL FERRITE, NICKEL-COBALT FERRITE, AND  
NICKEL-MANGANESE FERRITE USED IN THE ANNEALING STUDIES

| Code       | Composition  | Density and<br>Percent<br>of Theoretical | Grain<br>Size<br>( $\mu$ ) | $\kappa'$<br>X-Band | $\tan \delta$<br>X-Band | $h_{crit}$<br>(Oe)<br>KU-Band | $\chi''_{min}$<br>KU-Band | Remarks                         |
|------------|--|--|----------------------------|---------------------|-------------------------|-------------------------------|---------------------------|---------------------------------|
| 9696-5-7C  | NiFe <sub>2</sub> O <sub>4</sub>                                       | 5.36<br>99.6                             | 2.7                        | 14.1                | $1.5 \times 10^{-4}$    | 55                            | 0.018                     |                                 |
| 8354-76-7  | Ni <sub>0.977</sub> Co <sub>0.023</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.33<br>99.2                             | 4.1                        | 14.2                | $1.4 \times 10^{-4}$    | 68                            | 0.025                     |                                 |
| 9696-6-11C | Ni <sub>0.973</sub> Co <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.36<br>99.9                             | 3.2                        | 13.9                | $1.5 \times 10^{-4}$    | 97                            | 0.029                     |                                 |
| 9696-4-3   | Ni <sub>0.973</sub> Mn <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.36<br>99.6                             | 3.6                        | 14.6                | $3.4 \times 10^{-4}$    | 62                            | 0.018                     |                                 |
| 9696-5-5   | Ni <sub>0.973</sub> Co <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.38<br>99.9                             | 4.1                        | 14.6                | $3.4 \times 10^{-4}$    | 78                            | 0.027                     | 1st Mat'l<br>for<br>device test |
| 9696-10FS  | Ni <sub>0.973</sub> Co <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub> | 5.32<br>98.9                             | 4.0                        | 13.6                | $4.0 \times 10^{-4}$    | 89                            | 0.030                     | Flame<br>Sprayed                |

TABLE IV  
h<sub>crit</sub>, χ'', AND GRAIN SIZE AS A FUNCTION OF ANNEALING TEMPERATURE  
AND TIME FOR NICKEL, NICKEL-COBALT, AND NICKEL-MANGANESE FERRITES

| Code and<br>Composition  | Temp<br>(°C) | 0 min             |       |     | 30 min            |       |     | 60 min            |       |     | 300 min           |      |     | 960 min           |        |      |
|--|--------------|-------------------|-------|-----|-------------------|-------|-----|-------------------|-------|-----|-------------------|------|-----|-------------------|--------|------|
|  |              | h <sub>crit</sub> | χ''   | χ'' | h <sub>crit</sub> | G.S.  | χ'' | h <sub>crit</sub> | G.S.  | χ'' | h <sub>crit</sub> | G.S. | χ'' | h <sub>crit</sub> | G.S.   | χ''  |
| 9696-5-7C<br>NiFe <sub>2</sub> O <sub>4</sub>  | 1200         | 55                | 0.018 | 2.7 | 54                | ---   | --- | 43                | 0.007 | 3.6 | 43                | ---  | --- | 24                | ---    | 6.5  |
|  | 1250         | 55                | 0.018 | 2.7 | 38                | 0.007 | 5.5 | 34                | 0.007 | 6.5 | 24                | ---  | --- | 22                | ---    | 8.9  |
| 8354-76-7<br>Ni <sub>0.977</sub> Co <sub>0.023</sub> Fe <sub>2</sub> O <sub>4</sub>  | 1200         | 68                | 0.025 | 4.1 | 68                | ---   | --- | 68                | 0.011 | 5.3 | 68                | ---  | --- | 60                | ---    | 8.9  |
|  | 1250         | 68                | 0.025 | 4.1 | 68                | 0.022 | 7.6 | 68                | 0.023 | 8.9 | 61                | ---  | --- | 54                | ---    | 12.3 |
| 9696-6-11C<br>Ni <sub>0.973</sub> Co <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub> | 1200         | 97                | 0.029 | 3.2 | 86                | ---   | --- | 95                | 0.022 | 4.0 | 85                | ---  | --- | 76                | ---    | 6.5  |
|  | 1250         | 97                | 0.029 | 3.2 | 95                | 0.032 | 6.2 | 87                | 0.033 | 6.5 | 95                | ---  | --- | 77                | ---    | 10.5 |
| 9696-4-3<br>Ni <sub>0.973</sub> Mn <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub>   | 1200         | 62                | 0.018 | 3.6 | 31                | ---   | --- | 27                | 0.005 | 6.0 | 27                | ---  | --- | 24                | ---    | 8.3  |
|  | 1250         | 62                | 0.018 | 3.6 | 27                | 0.007 | 7.4 | --                | 0.005 | 9.9 | 27                | ---  | --- | 24                | ---    | 11.0 |
| 9696-5-5<br>Ni <sub>0.973</sub> Co <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub>   | 1200         | 78                | 0.027 | 4.1 | 70                | 0.023 | --- | 61                | 0.020 | --- | --                | ---  | --- | ---               | ---    | ---  |
|  | 1100         | 78                | 0.027 | 4.1 | --                | ---   | --- | --                | ---   | --- | --                | ---  | --- | 55                | 0.020* | ---  |
| 9696-10FS<br>Ni <sub>0.973</sub> Co <sub>0.027</sub> Fe <sub>2</sub> O <sub>4</sub>  | 1200         | 89                | 0.030 | 4.0 | 87                | 0.032 | --- | --                | ---   | --- | --                | ---  | --- | --                | ---    | ---  |

\* 5280 minutes

while the critical field has remained unchanged. This value of magnetic loss is comparable to a conventionally prepared material and gives this approach considerable encouragement. The threshold field data on one cobalt composition (9696-6-11C) are somewhat scattered. Separate spheres cut from a single hot-pressed disk were annealed and measured, for each time, and the scatter is probably due to the variation in  $h_{crit}$  throughout the disk. This, unfortunately, makes it difficult to make unambiguous conclusions about the effects of annealing on microwave properties.

The results obtained on the samples being tested in the device (9696-5-5) are also contained in this table. After 5280 minutes at  $1100^{\circ}\text{C}$ , both the magnetic loss and critical field have been reduced by about 25 percent. It may be that a shorter anneal at a higher temperature will produce the desired results of reducing  $\chi''$  while maintaining a high  $h_{crit}$  as was possible with the previous cobalt sample, 8354-76-7. The results on the one flame-sprayed material are not complete enough for comment, and additional data will be obtained during the final quarter.

#### 4.6 Device Development

##### 4.6.1 Device requirements

The device to be considered under this contract is a four-port differential phase-shift circulator with design goals of 1.5 Mw and 1.5 kw average power,  $\leq 0.5$  dB insertion loss, to operate from 16 to 17 kMc in WR62 KU-band waveguide. It is of prime interest to compare the performance of a conventionally-fired ferrite with a hot-pressed fine-grain ferrite in such a device application and thus establish to what extent the state-of-the-art at high power is improved by using fine-graining techniques. Construction of single  $90^{\circ}$  differential phase-shift section exhibiting  $\leq 0.5$  dB insertion loss at the design goal power level of 750 kw/750 w will be acceptable as a convincing demonstration of device capability, and a complete four-port circulator will not be constructed under this contract.

#### 4.6.2 Estimate of material requirements

The general ferrite material properties required for a low-loss below-resonance differential phase-shifter include a small  $\gamma \cdot \Delta H$  product, low  $\tan \delta$ , low  $\chi''$ , and a high Curie temperature. Estimates of insertion loss and critical power level as a function of material properties can be made using the theoretical work of Suhl and Schlömann. In making such estimates, a number of assumptions and approximations must be made which introduce an unknown factor of uncertainty into the estimate.

In standard WR62 KU-band waveguide the field strength at the plane of circular polarization can readily be calculated. At 16.5 Gc and for an incident power of 750 (i. e. , 1.5 Mw input to a complete circulator) the rf magnetic field in the phase-shift waveguide at the plane of c. p. is 52 Oe. In differential phase-shift circulator design a flat slab of ferrite is placed along the broad face of the waveguide crossing the plane of c. p. The first assumption is that all the material is at the plane of c. p. while in actuality it extends in both directions into elliptically polarized fields. In usual practice the external biasing field is applied perpendicular to the slab just sufficient in magnetiude to magnetize the material. From Suhl's theory the threshold field can be calculated for a similar geometry, a thin disk with the external field applied perpendicular to the plane of the disk such that the internal field is equal to zero.

The result is

$$\left( \frac{h_{\text{crit}}^{\text{c. p.}}}{\Delta H_K} \right) \text{ disk } H_i = 0 = 2.10 \quad (1)$$

at 16.5 Gc for material with a magnetization of 3000 gauss. The high power material testing is performed in a cavity on a sphere using linear polarization (l p. ) and with external biasing field ( $H_{\text{ex}}$ ) of 1500 Oe. The result for this configuration is

$$\left( \frac{h_{\text{crit}}^{\text{l. p.}}}{\Delta H_K} \right)_{\text{sphere } H_{\text{ex}} = 1500 \text{ Oe}} = 3.00 \quad (2)$$

at 16.5 Gc for a material with a magnetization of 3000 gauss. The second assumption is that  $\Delta H_K$  is the same for both situations. Then

$$\begin{aligned} h_{\text{crit}}^{\text{l. p.}} (\text{l. p. sphere } H_{\text{ex}} = 1500 \text{ Oe}) &= \frac{3.00}{2.10} \times h_{\text{crit}}^{\text{c. p.}} (\text{disk } H_i = 0) \\ &= \frac{3.00}{2.10} \times 52 \text{ Oe} = 74 \text{ Oe} \quad (3) \end{aligned}$$

The estimate of the insertion loss can be made using a calculation by Schlömann.<sup>2</sup> The insertion loss,  $(L_{\pi/2})$ , of a thin slab of material located at the plane of circular polarization for 90° of phase shift is given by

$$L_{\pi/2}(\text{dB}) = \frac{4.343 \pi}{2} \frac{\chi'' + \epsilon''/\epsilon'}{K'}^2 \quad (4)$$

Note that once again it has been assumed that all the material is at the plane of c. p. This equation can be simplified by virtue of the low dielectric loss, i. e.,  $\epsilon''/\epsilon'^2 \ll \chi''$ , and by putting

$$K' = \frac{(\gamma 4\pi M)}{\omega} \quad (5)$$

Then

$$L_{(\pi/2)}(\text{dB}) = \frac{4.343 \pi \chi'' \omega}{2 (\gamma 4\pi M)} \quad (6)$$

Taking a value of 3000 gauss for  $4\pi M$  and  $\omega = 16.5 \text{ Gc}$  we get

$$L_{(\pi/2)}(\text{dB}) = 12.6 \chi''$$

From this theoretical relationship, the material magnetic loss requirement for the device can be estimated. The achievement of a 0.5 dB insertion loss for the complete circulator necessitates 0.4 dB loss in the phase shift section (this allows 0.1 dB for flanging leakage losses, etc.).

$$\begin{aligned}\chi'' &\leq \frac{\text{insertion loss in dB}}{12.6} \\ &\leq \frac{0.4}{12.6} \leq 0.03\end{aligned}\tag{7}$$

It is important to note that several assumptions are made in the theoretical process of relating circulator threshold level and sphere  $h_{\text{crit}}$ . Evidence has been obtained previously indicating that the threshold levels observed in practice are significantly lower than predicted from the theory. At present it appears that the most questionable factors involved are the assumptions that  $\Delta H_K$ , the spin-wave linewidth, is the same for different geometries at different values of  $H_{\text{dc}}$ , and that all of the ferrite material in the waveguide is exposed to a purely circularly polarized driving wave. It is, therefore, stressed that the predicted threshold power levels given in this report are theoretically optimum values which are unlikely to be achieved in practice if previous experience is any guide. This experience suggests that the threshold power level in an actual device may be as low as one-half of the theoretical value. An important part of the device program on this contract will be to obtain high-power data in waveguide to establish a practical working relationship between theory and practice.

#### 4.6.3 Low-power studies

##### 4.6.3.1 Conventionally-fired nickel ferrite

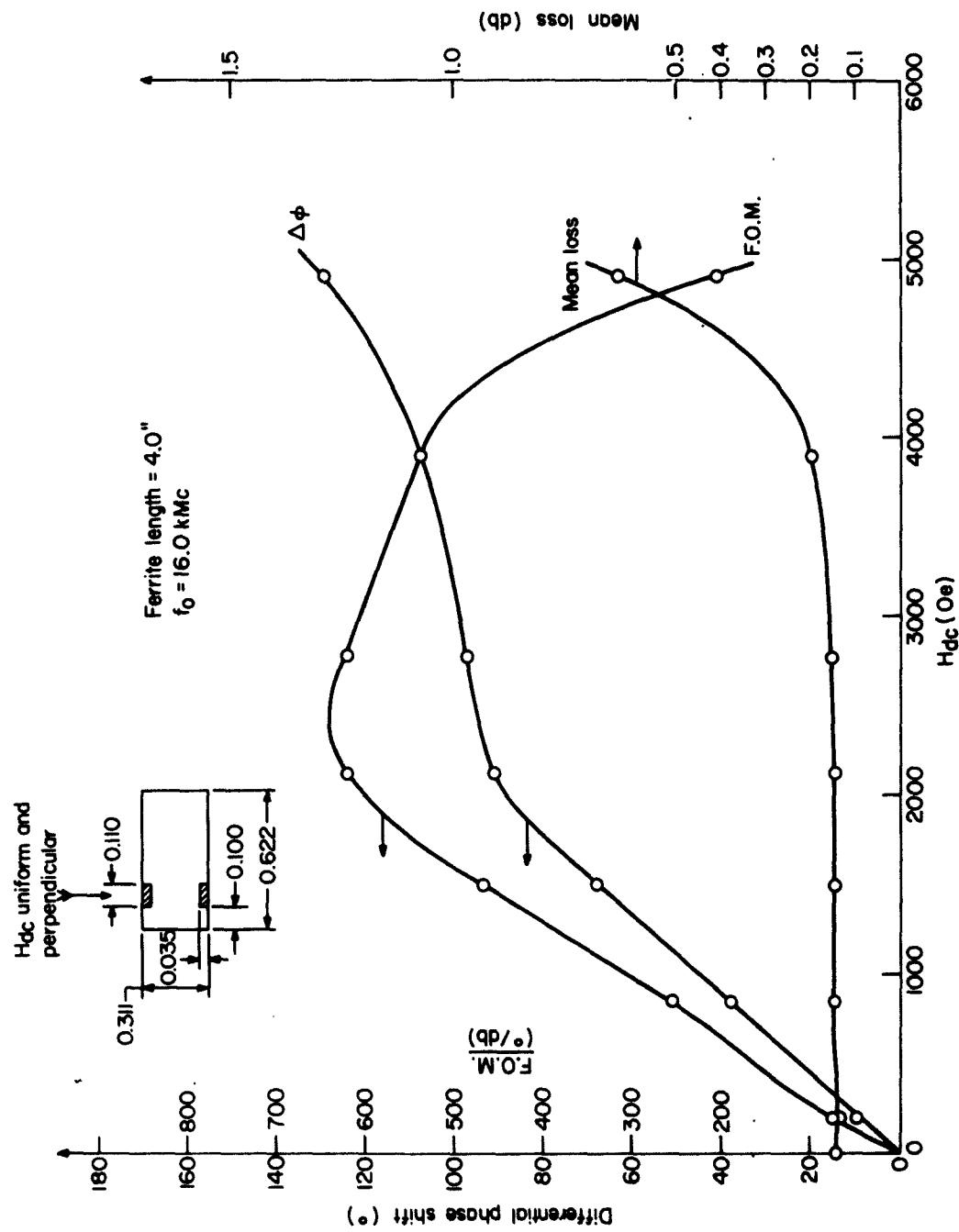
In the interest of making the most valid comparison between conventionally-fired and hot-pressed fine-grain nickel ferrite a material

was selected having a similar basic composition. Since the most promising of the hot-pressed materials was a Ni-Co composition, a similar Ni-Co material was selected from the several available conventionally-fired ferrites. Raytheon R-192 Ni-Co ferrite which is a well-established commercial material was selected for the comparison. The properties of the particular batch of R-192 used are shown in Table V.

Geometry optimization studies were conducted using the R-192 material to establish a ferrite/waveguide geometry appropriate to the device under consideration. The desired features of the geometry included:

- a. High figure of merit, ( $^{\circ}/\text{dB}$ ), to obtain low insertion loss,
- b. A thin, wide H-plane slab to minimize breakdown problems due to the peak power and excessive temperature gradient across the ferrite thickness due to the high average power,
- c. Deviation of differential phase-shift of  $< \pm 10^{\circ}$  about  $90^{\circ}$  over the frequency band, and freedom from resonances and excessive dispersion at the upper frequencies,
- d. Reasonable physical length and operating  $H_{\text{dc}}$ .

A highly satisfactory device geometry evolved from the work. This is shown in Fig. 1 along with plotted data for insertion loss, differential phase-shift, and figure of merit at low-power at room temperature. It can be seen that the "knee" of saturation occurs for an applied  $H_{\text{dc}}$  of the order 2500 Oe where the figure of merit is approximately  $600^{\circ}/\text{dB}$ . Allowing 0.1 dB additional loss in castings, flanges, and leakage, it may be predicted that a complete circulator using the R-192 material in this geometry would exhibit an over-all insertion loss of  $\leq 0.3$  dB. From the  $h_{\text{crit}}$  of 12.3 Oe a  $P_{\text{crit}}$  of the order of 50 kw peak is theoretically predicted for such a circulator. The data shown in Fig. 1 are at 16.0 kMc. The excellent bandwidth properties obtained for the geometry are such that variations of loss and phase-shift over the frequency band are insignificant from the device point-of-view. The data shown are thus accurately representative of the entire frequency band.



LOW POWER PERFORMANCE OF R-192 PHASE-SHIFTER  
FIGURE 1

TABLE V  
PROPERTIES OF THE CONVENTIONAL AND HOT-PRESSED FERRITES  
USED IN THE DEVICE TESTING

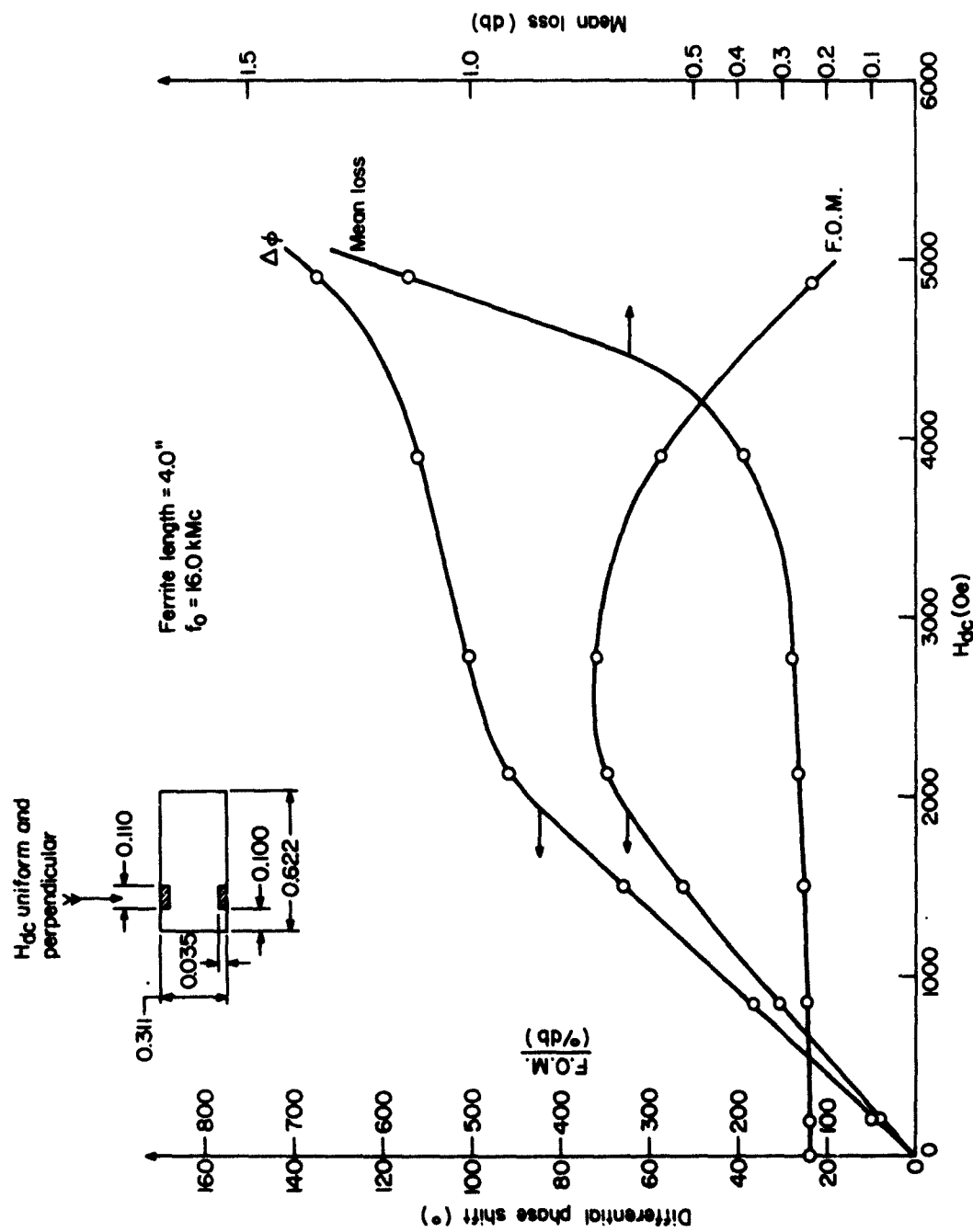
| Property             | R-192<br>Batch 919   | Hot-Pressed<br>Ni-Co Ferrite |
|----------------------|----------------------|------------------------------|
| $4\pi M_s$ (gauss)   | 2845                 | 3242                         |
| $\Delta H_x$ (Oe)    | 218                  | 417                          |
| $\gamma(Mc/Oe)$      | 3.2                  | ---                          |
| $\tan \delta$        | $2.3 \times 10^{-4}$ | $3.4 \times 10^{-4}$         |
| $h_{crit}^*$         | 12.3                 | 77.7                         |
| $\chi''$             | 0.014                | 0.027                        |
| $K'$                 | 13                   | 14.6                         |
| Density              | 98%                  | 99.9%                        |
| Grain Size ( $\mu$ ) | 12.3                 | 4.1                          |

\* Measured for sphere at  $H_{dc} = 1500$  Oe (i.e., below SR)

#### 4. 6. 3. 2 Hot-pressed fine-grain nickel ferrite

The composition  $\text{Ni}_{0.973}\text{Co}_{0.027}\text{Fe}_2\text{O}_4$  was chosen as the material for initial device testing because its over-all properties correspond closely to the above theoretical estimates of insertion loss and threshold level. Batch 9696-5-5 prepared by hot pressing was used. The properties of this composition when first pressed are shown in Table V. A total of 5 one-half inch diameter disks were pressed and carefully sliced to yield samples for  $\tan \delta$ ,  $\chi''$ , and  $h_{\text{crit}}$  measurements and several small pieces having the device cross section. The  $h_{\text{crit}}$  and  $\chi''$  for each disk were evaluated and are included in Table VI. The 4-inch ferrite length in the experimental phase-shift was thus made up of 9 small pieces cemented end-to-end. The geometry used was the same as the R-192 geometry referred to above.

Data obtained at low power and room temperature is shown in Fig. 2. As in the case of the R-192 phase-shifter, no significant variation with frequency was observed and the data are again shown at a single frequency only. The differential phase-shift obtained for the hot-pressed material is essentially the same as the R-192 material. The insertion loss is seen to be higher by a factor of the order of 2. This is as expected because, with negligible dielectric loss contribution (i. e. , low  $\tan \delta$ ),  $\chi''$ , the magnetic susceptibility, is the primary loss component. Since  $\chi''$  for the hot-pressed material is a factor of 2 higher than for the R-192 material, the insertion loss is expected to increase by the same factor. Taking a figure of merit of  $300^\circ/\text{dB}$  and allowing the same 0.1 dB additional loss incurred in a complete circulator leads to the prediction that a circulator using the hot-pressed Ni-Co ferrite would exhibit an over-all insertion loss  $< 0.5$  dB. From the average  $h_{\text{crit}}$ 's, a  $P_{\text{crit}}$  slightly in excess of 1.5 Mw peak is theoretically predicted for such a circulator. A limited investigation of temperature dependence has indicated that no significant variation in low-power performance occurs up to  $60^\circ\text{C}$ .



LOW POWER PERFORMANCE OF HOT - PRESSED NI-CO FERRITE (9696-5-5)  
PHASE - SHIFTER

FIGURE 2

TABLE VI  
PROPERTIES OF FINE HOT-PRESSED DISKS OF NICKEL-COBALT FERRITE  
(9696-5-5) USED IN DEVICE TESTING

| Disk Code  | $\chi''$ | $\frac{h_{crit}}{Oe}$<br>KU-Band |
|------------|----------|----------------------------------|
| 9696-5-5-H | 0.035    | 76                               |
| 9696-5-5-I | 0.028    | 85                               |
| 9696-5-5-J | 0.027    | 76                               |
| 9696-5-5-G | 0.027    | 68                               |
| 9696-5-5-D | 0.030    | 85                               |

#### 4.6.4 High-power studies

At the time of writing this report no high-power data had been obtained on either of the two ferrite materials being studied. A WR-62 resonant ring test facility has been designed, all parts procured, and preliminary construction started. The ring has been designed to provide at least the 750 kw peak power required to test experimental phase-shifters to the device design goals of the contract. It is anticipated that the use of the electro-negative gas  $\text{SF}_6$  will be necessary to prevent breakdown and provision has been made for this in the test facility. Based on the theoretical estimates using  $h_{\text{crit}}$  data for spheres, it is predicted that an improvement in threshold power of the order 30:1 should be observed for the hot-pressed material. It will be highly interesting to check this prediction by actual measurements.

#### 4.6.5 Summary

A satisfactory device geometry has been established for the nickel ferrites under investigation. Using the conventionally-fired R-192 Ni-Co ferrite, a circulator with  $\leq 0.3$  dB insertion loss at low power has been established with a threshold power level of the order 50 kw peak theoretically predicted. The same circulator using hot-pressed fine-grain Ni-Co ferrite would have a higher low-power insertion loss of  $\leq 0.5$  dB, due to a higher  $\chi''$ , with a threshold power in excess of 1.5 Mw theoretically predicted. No high-power data have been obtained at the time of writing this report. Attention is drawn to the comments made in Sec. 4.6.2 in reference to the expected discrepancy between theoretical and practical threshold levels, and the necessity of high-power measurements to determine the extent of these derivations.

## 5.0 CONCLUSIONS

Hot-pressed fine-grain ferrites have threshold fields up to five times those of conventionally prepared ferrites, and low-power magnetic losses about two to three times greater than the latter. Losses of this magnitude are sufficiently low to meet the low-power specifications called for in the device being considered for this program. However, the ultimate objective in advancing the state-of-the-art should consist of increasing the power capability of ferrite phase-shift devices while maintaining the losses close to the levels attainable with conventional materials. To this end, studies have been directed at lowering the magnetic losses in high threshold field materials. These studies have consisted of searching for combinations of grain size, hot-pressing temperature, composition, and annealing procedure that would meet the above objectives.

## 6.0 PLANS FOR NEXT QUARTER

1. Annealing studies will be continued on those compositions listed in Table IV. If time permits other compositions will be annealed, especially those with high  $h_{crit}$ 's.

2. Additional flame-sprayed powders will be prepared and hot pressed. Preliminary results reported this quarter on one flame-sprayed, hot-pressed sample indicate that high  $h_{crit}$  materials with losses comparable to ball-milled, hot-pressed materials can be made.

3. Some effort will be made to determine grain size distributions, which should provide a better characterization of the microstructures and the relationship to threshold fields.

4. Longer hot-pressing times will be employed in an attempt to lower magnetic losses.

5. The WR-62 resonant ring facility will be completed early in the next quarter, and high-power insertion loss data for the conventionally-fired and hot-pressed fine-grain Ni-Co ferrites will be obtained in a practical device geometry. Should the initially tested fine-grain material not come up to expectations, another composition with a significantly higher  $h_{crit}$  and comparable  $\chi''$  will be chosen.

## 7.0 IDENTIFICATION OF PERSONNEL

Below is a list of the personnel and the time spent on this project from 1 January 1965 - 31 March 1965.

|              | <u>Classification</u>        | <u>Hours</u> |
|--------------|------------------------------|--------------|
| S. Waugh     | Senior Research Scientist    | 292          |
| A. Paladino  | Principal Research Scientist | 112          |
| J. Green     | Principal Research Scientist | 28.5         |
| B. Healy     | Associate Research Scientist | 364          |
| S. Georgian  | Research Assistant           | 106          |
| S. Cvikevich | Research Scientist           | 4            |
| A. Booth     | Senior Engineer              | 25           |
| M. Armstrong | Engineer                     | 180          |
| T. Quinlan   | Engineer                     | <u>3</u>     |
|              | Total                        | 1114.5       |

Michael J. Armstrong - Engineer

Upon completion of a student apprenticeship in radio engineering with Philips Electrical Ltd. , Mr. Armstrong took a degree course at Imperial College of Science and Technology, London University, where he received a B.S. in physics in 1960.

From 1960 to 1964 Mr. Armstrong was engaged in magnetron development and production at the Mullard Radio Valve Company, England.

In November 1964 Mr. Armstrong came to the United States to join Raytheon Company, Special Microwave Devices Operation, where he is mainly concerned with research and development of high-power differential phase-shift circulators.

Mr. Armstrong is an associate member of the IEE, London, England.

Thomas E. Quinlan - Engineer

In 1957 Mr. Quinlan received his B.S. in marine and electrical engineering from Massachusetts Maritime Academy. From 1958 to 1960 he did graduate work at Northeastern University.

From 1958 to 1960 he was engaged in research and development of microwave components at Bomac Labs, Inc. Beverly, Massachusetts. From 1959 to 1962 he was employed by Raytheon Company, Power Tube Division, doing pilot production work on magnetrons and klystrons. Since 1962 Mr. Quinlan has been engaged in development of ferrite devices at the Special Microwave Devices Operation of Raytheon Company.

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| 13. ABSTRACT<br>A number of new compositions were prepared in the vicinity of the compensation point in the series $Ni_{1-x}Co_xFe_2O_4$ , with $0 \leq x \leq 0.04$ . No systematic variation in the threshold field or magnetic loss was observed with cobalt concentration, although threshold fields were as high as 108 Oe at KU-band. Lowering the hot-pressing temperature from 1200° to 1150°C had no consistent effect on microwave properties. Annealing studies at 1200° and 1250°C indicate that the threshold field decreases significantly with time for nickel and nickel-manganese ferrites, whereas it remains high in nickel-cobalt ferrites. The magnetic loss in general decreases with increasing time; it decreases most for nickel and nickel-manganese ferrites, and least for nickel-cobalt ferrites.<br>A satisfactory device geometry has been established for a differential phase-shift circulator. Low power phase shift and insertion loss measurements have been made on a conventional and fine-grain, nickel-cobalt ferrite. Although the insertion loss of the fine-grain ferrite is about double that of the conventional material due to a magnetic loss of approximately twice that of the conventional material, a circulator with $\leq 0.5$ dB loss can be constructed. |                              |   |

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|    | <p>Ferrites (Fine Grain)</p> <p>High Power Microwave Device</p> <p>Ku Band</p> <p>Nickel Cobalt Ferrite</p> |        |    |        |    |        |    |

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